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Reduction of the Unique Carbonyl in $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-CCO})$ by $BH_3\cdot THF$. Molecular Structure of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$

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Introduction

Terminal carbon monoxides of metal carbonyls have been reduced by strong reducing agents such as AlH_3, $LiAlH_4$, and $LiHBEt_3$.[1−3] While diborane in THF ($BH_3\cdot THF$) is not effective for such reduction reactions, Masters[4] found that $BH_3\cdot THF$ can reduce iron and ruthenium acetyl complexes quantitatively to alkyl complexes under mild conditions. The unique carbonyl of the borylidyne cluster $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-BCO})$[5] is also readily reduced by BH_3 in THF. In this case a methylene group is formed, giving rise to $(\mu\text{-H})_3Os_3(CO)_9\text{-}(\mu_3\text{-}\eta^2\text{-BCH}_2)$,[6] a vinylidyne cluster analogue (eq 1). This reaction appears to

$$(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-BCO}) + BH_3\cdot THF \rightarrow (\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-BCH}_2) + {}^1/_3(HBO)_3 + THF \tag{1}$$

occur through addition of BH_3 to the oxygen of the (μ_3-BCO) unit, the most negatively charged oxygen in the molecule.[7] This step, followed by transfer of two hydrogen atoms to the carbon atom with subsequent elimination of HBO, would leave behind $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-BCH}_2)$. Since the ketenylidene cluster $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-CCO})$[8] is isoelectronic with $(\mu\text{-H})_3Os_3(CO)_9\text{-}(\mu_3\text{-BCO})$, it is of interest to us to determine to what extent its reaction with BH_3 parallels that of the borylidyne cluster.

Results and Discussion

Synthesis of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$. Treatment of $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-CCO})$ with excess $BH_3\cdot THF$ in CH_2Cl_2 at room temperature results in reduction of the unique carbonyl to a methyl group, giving the ethylidyne cluster $(\mu\text{-H})_3Os_3(CO)_9\text{-}(\mu_3\text{-CCH}_3)$[9−12] in yields up to 61%. NMR spectroscopy was employed to follow the reaction. When fresh $BH_3\cdot THF$ that was prepared in this laboratory was used, with or without 5% $NaBH_4$ stabilizer, no evidence was observed for the formation of intermediate species, such as the vinylidene cluster $(\mu\text{-H})_2Os_3\text{-}(CO)_9(\mu_3\text{-}\eta^2\text{-C}{=}CH_2)$.[13−15] However, when commercially available $BH_3\cdot THF$ was employed, trace amounts of the vinylidene cluster were detected in proton NMR spectra of solutions allowed to stand at room temperature for several days.

From boron-11 NMR spectra of samples that were obtained from freshly prepared $BH_3\cdot THF$ without $NaBH_4$ stabilizer, a signal was observed at 4.1 ppm. This material was not identified. A single-crystal X-ray structure determination verified the formation of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$, and its NMR and IR spectra are in accord with literature reports of ^1H NMR[9−11] and IR[11] spectra.

Attempted Reduction of $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-C}{=}CH_2)$ by $BH_3\cdot THF$. In view of the reduction of $(\mu\text{-H})_3Os_3(CO)_9\text{-}(\mu_3\text{-BCO})$ by $BH_3\cdot THF$ to form $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-BCH}_2)$,[6] the possibility was considered that $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-C}{=}CH_2)$ might be an intermediate in the conversion of $(\mu\text{-H})_2\text{-}Os_3(CO)_9(\mu_3\text{-CCO})$ to $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$ in the reaction with $BH_3\cdot THF$. However, treatment of $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-C}{=}CH_2)$ with pure, freshly prepared $BH_3\cdot THF$, with or without 5% $NaBH_4$ for 5 days in CH_2Cl_2, yielded no $(\mu\text{-H})_3Os_3(CO)_9\text{-}(\mu_3\text{-CCH}_3)$. When commercially available $BH_3\cdot THF$ was employed, only trace amounts of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$ were detected by NMR spectroscopy. While it is likely that the reduction of $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-CCO})$ involves initial attachment of BH_3 to the unique carbonyl followed by transfer of hydrogen to the carbon, it is unlikely that $(\mu\text{-H})_2Os_3(CO)_9\text{-}(\mu_3\text{-}\eta^2\text{-C}{=}CH_2)$ is formed as a discrete intermediate in the conversion of $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-CCO})$ to $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$.

Synthesis of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-}^{13}\text{CCH}_3)$. The reaction of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-BCO})$ with BCl_3 produces an interchange of boron and carbon atom positions of the μ_3-BCO unit in the formation of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CBCl}_2)$.[16] This result raises the possibility that the reaction of $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-CCO})$ with $BH_3\cdot THF$ might proceed through an interchange of the carbon atom positions of the μ_3-CCO unit. To test this possibility, the reaction of selectively labeled $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-}^{13}\text{CCO})$[8] with $BH_3\cdot THF$ was examined. The only product observed by NMR spectroscopy[9−12] is $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-}^{13}\text{CCH}_3)$. The ^{13}C NMR spectrum of the product consists of a singlet at 154.7 ppm which is assigned to the capping carbon. Thus there is no apparent interchange of carbon atoms in the reaction.

Structure of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$. Light yellow crystals of $(\mu\text{-H})_3Os(CO)_9(\mu_3\text{-CCH}_3)$ suitable for a single-crystal X-ray analysis were grown by slow evaporation from $CHCl_3$ at room temperature, and its molecular structure was determined (Figure 1). Selected bond distances and bond angles are given in Table 1.

The $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$ molecule is isostructural with $(\mu\text{-H})_3(CO)_9Ru_3(\mu_3\text{-CCH}_3)$[17] and $(\mu\text{-H})_3Fe_3(CO)_9(\mu_3\text{-CCH}_3)$.[18] The structure consists of an Os_3 triangle with three terminal

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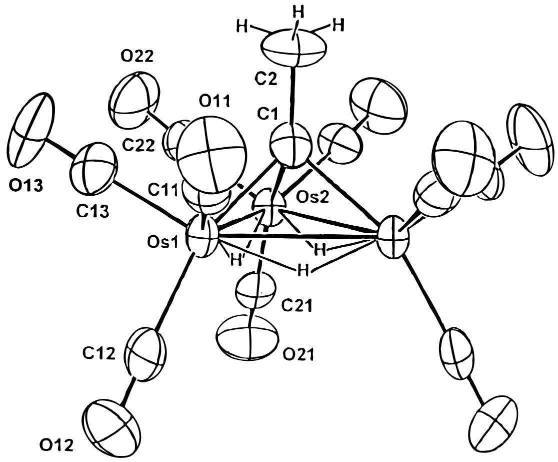

Figure 1. ORTEP plot of the molecular structure of $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$ with 50% probability ellipsoids.

Table 1. Selected Bond Distances (Å) and Angles (deg) for $(\mu\text{-H})_3Os_3(CO)_9(\mu_3\text{-CCH}_3)$

Bond Distances			
Os1−Os1′	2.878(1)	Os2−C21	1.91(3)
Os1−Os2	2.875(1)	Os2−C22	1.89(2)
Os1′−Os2	2.875(1)	C1−C2	1.48(3)
Os1−C1	2.08(2)	C11−O11	1.11(2)
Os1−C11	1.93(2)	C12−O12	1.15(2)
Os1−C12	1.96(2)	C13−O13	1.12(2)
Os1−C13	1.91(2)	C21−O21	1.20(3)
Os2−C1	2.07(2)	C22−O22	1.11(2)
Angles			
Os1′−Os1−Os2	59.97(1)	Os1−Os2−C1	46.4(5)
Os1−Os1′−Os2	59.97(1)	Os1−Os2−C21	118.2(5)
Os1′−Os2−Os1	60.07(3)	Os1−Os2−C22′	142.8(5)
Os1′−Os1−C1	46.3(5)	Os1−Os2−C22	95.2(5)
Os1′−Os1−C11	95.7(5)	C1−Os2−C21	160.2(9)
Os1′−Os1−C12	116.8(5)	C1−Os2−C22	96.5(7)
Os1′−Os1−C13	144.7(5)	C21−Os2−C22	97.6(6)
Os2−Os1−C1	46.0(6)	C22−Os2−C22′	90(1)
Os2−Os1−C11	142.9(5)	Os1−C1−Os1′	87(1)
Os2−Os1−C12	120.7(5)	Os1−C1−Os2	87.6(8)
Os2−Os1−C13	95.2(5)	Os1−C1−C2	128(1)
C1−Os1−C11	96.9(8)	Os2−C1−C2	126.(2)
C1−Os1−C12	160.4(7)	Os1−C11−O11	176.(2)
C1−Os1−C13	98.5(7)	Os1−C12−O12	179.(2)
C11−Os1−C12	94.7(7)	Os1−C13−O13	177.(2)
C11−Os1−C13	90.9(7)	Os2−C21−O21	179.(2)
C12−Os1−C13	97.1(7)	Os2−C22−O22	178.(2)

carbonyls bound to each Os atom. It has apparent C_{3v} symmetry. The triosmium framework is symmetrically capped by a methylidyne carbon bearing a methyl group. The ethylidyne C−C bond is perpendicular to the Os_3 plane with a tilt angle of 0.6° from perpendicularity. For the methylidyne clusters $(\mu\text{-H})_3Os(CO)_9(\mu_3\text{-CR})$ (R = Cl, Br, C_6H_5) tilt angles of 0.1, 0.2, and 3.0, respectively, have been observed.[19] Positions of the hydrogen atoms were not directly located; however, they are inferred as bridging hydrogens across the basal osmium−osmium bonds based on the ^{1}H-NMR chemical shift (−18.68 ppm)[8a] and comparison of the Os−Os distances with the previously reported values for Os−H−Os distances.[8b] The present structure is consistent with an approximate structure of $(\mu\text{-H})_3Os(CO)_9(\mu_3\text{-CCH}_3)$ that was deduced from an X-ray powder diffraction analysis.[20]

Experimental Section

Materials. Prepurified H_2 gas (AGA Inc.) was used as received. $BH_3\cdot THF$ (1.0 M in THF) and Diazald (99% ^{13}C) (Aldrich Chemical Co.) for the preparation of diazomethane were used as received. $NaBH_4$ (Aldrich Chemical Co.), was heated over night to 45 °C under dynamic vacuum before use. Diazomethane was prepared according to the method described in ref 21. B_2H_6 was prepared according to a method in the literature[22] and stored at −196 °C in a Pyrex glass tube equipped with a ground glass stopcock prior to use. $Os_3(CO)_{12}$ (Strem Chemical Co.) was used as received. $Os_3(^{13}CO)_{12}$ was prepared according to a previously described procedure.[23] $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-CCO})$ and $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-}^{13}\text{CCO})$,[8] $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-}\eta^2\text{-C=CH}_2)$,[13,14] and $Os_3(CO)_{11}(MeCN)$[24] were prepared and isolated by methods described in the literature.

Table 2. Crystallographic Data for $(\mu\text{-H})_3Os(CO)_9(\mu_3\text{-CCH}_3)$

formula	$C_{11}H_6O_9Os_3$
fw	852.77
space group	*Pnma* (No. 62)
a, Å	17.532(3)
b, Å	14.573(6)
c, Å	6.670(2)
vol, Å^3	1704.1
Z	4
ρ(calcd), g cm^{-3}	3.324
radiation	Mo Kα (λ = 0.710 73 Å)
μ(Mo Kα), cm^{-1}	223.9
max transm, %	99.98
min transm, %	46.05
T, °C	25
scan mode	$\omega-2\theta$
2θ limits	4−45°
±*h*	−18, 18
±*k*	−15, 15
±*l*	0, 7
no. of reflcns collcd	4922
no. of unique reflcns [$I \geq 3.0\sigma(I)$]	806
no. of variables	113
$R_F{}^a$	0.025
$R_{wF}{}^b$	0.032
k^c	0.04

^{a} $R_F = \sum||F_o| - |F_c||/\sum|F_o|$. ^{b} $R_{wF} = \{\sum w[|F_o| - |F_c|]^2/\sum w|F_o|^2\}^{1/2}$. ^{c} $w = (\sigma(I)^2 + (kI)^2)^{-1}$.

X-ray Crystal Structure Determination. Crystallographic data were collected at room temperature on an Enraf-Nonius CAD4 diffractometer with graphite-monochromated Mo Kα radiation. All the crystallographic computations were carried out on a DEC Vax Station 3100 computer using MolEN.[25] Unit cell parameters were obtained by a least-squares refinement of the angular settings from 25 reflections, well distributed in reciprocal space and lying in a 2θ range of 24−30°. All reflection data were corrected for Lorentz and polarization effects. An empirical absorption correction was applied based on measured ψ scans. Crystallographic data are summarized in Table 2.

The structure was solved by the direct method MULTAN 11/82 and difference Fourier synthesis with analytical atomic scattering factors used throughout the structure refinement with both the real and imaginary components of the anomalous dispersion included for all non-hydrogen atoms. Full-matrix least square refinements were employed. After Os atoms were located and refined, all non hydrogen atoms were located from the difference Fourier maps. One Os atom, one oxygen atom, and three carbon atoms are on a mirror plane at $y = {}^1/_4$. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions on the methyl group were calculated. Positional and isotropic displacement parameters of these hydrogen atoms were fixed during the refinement. Final atomic positional parameters are listed in Table 3.

Reaction of $(\mu\text{-H})_2Os_3(CO)_9(\mu_3\text{-CCO})$ with $BH_3\cdot THF$. An NMR tube joined to an adaptor and attached to the vacuum line was charged

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Table 3. Positional Parameters and Their Esd's for $(\mu\text{-H})_3\text{Os}(\text{CO})_9(\mu_3\text{-CCH}_3)$

atom	x	y	z	B,[a] Å^2
Os1	0.11179(3)	0.34874(3)	0.70275(9)	2.77(1)
Os2	0.25373(5)	0.250	0.6957(1)	2.63(2)
C1	0.161(1)	0.250	0.888(3)	3.3(4)
C2	0.165(2)	0.250	1.110(4)	5.7(7)
C11	0.0178(9)	0.362(1)	0.851(2)	3.8(4)
O11	−0.0340(7)	0.3674(9)	0.946(2)	6.2(3)
C12	0.073(1)	0.409(1)	0.461(2)	4.1(4)
O12	0.0509(9)	0.4435(8)	0.318(2)	7.4(4)
C13	0.1527(9)	0.456(1)	0.828(2)	4.1(4)
O13	0.1802(9)	0.5162(8)	0.902(2)	8.1(4)
C21	0.312(1)	0.250	0.452(3)	3.3(5)
O21	0.349(1)	0.250	0.302(3)	6.2(5)
C22	0.3100(9)	0.159(1)	0.832(2)	3.7(3)
O22	0.3434(8)	0.1066(9)	0.918(2)	6.9(3)

[a] Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

with a THF-d_8 solution of $(\mu\text{-H})_2\text{Os}_3(\text{CO})_9(\mu_3\text{-CCO})$ (0.0095 mmol) and cooled to −196 °C. While at this temperature, B_2H_6 (0.0095 mmol) was condensed onto the solution and the NMR tube sealed under vacuum. The NMR sample was warmed to room temperature and the progress of the reaction was followed at room temperature by ^{1}H and ^{11}B NMR over a period of 16 h. Conversion from $(\mu\text{-H})_2\text{Os}_3(\text{CO})_9(\mu_3\text{-CCO})$ to $(\mu\text{-H})_3\text{Os}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$ was 50−55%. The ^{11}B NMR spectrum indicated formation of a boron oxide species[12] (4.1 ppm) which was not identified.

$(\mu\text{-H})_3\text{Os}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$ was isolated in 61% yield (55 mg) from a solution of $(\mu\text{-H})_2\text{Os}_3(\text{CO})_9(\mu_3\text{-CCO})$ (90 mg, 0.104 mmol) and B_2H_6 (1.0 mmol in 5 mL THF) in 30 mL CH_2Cl_2 that was stirred for 1 h at room temperature. Volatile components were removed and the resulting yellow residue was purified by TLC (2 mm silica gel, 1:4 toluene: hexane). ^{1}H NMR (CDCl_3, 30 °C): δ 4.43 (s, 3H), −18.68 (s, 3H) ppm. IR (CHCl_3): ν_{CO} 2077 m, 2068 m, sh, 2056 m, 2033 s, sh, 2021 s cm^{-1}. These spectra are in accord with earlier reports for $(\mu\text{-H})_3\text{Os}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$.[9−11] The mass spectrum contains a parent ion peak that corresponds to the molecular formula $^{12}\text{C}_{11}{}^{1}\text{H}_6{}^{16}\text{O}_9{}^{192}\text{Os}_3$: m/z (obsd) 858; m/z(calcd) 858. The sequential loss of each of the nine carbonyls from the molecular ion is visible in the mass spectrum.

Similar yields of $(\mu\text{-H})_3\text{Os}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$ were obtained when the $\text{BH}_3\cdot\text{THF}$ contained 5% NaBH_4 and when commercial $\text{BH}_3\cdot\text{THF}$ was employed. On occasion, however, trace amounts of the vinylidene cluster $(\mu\text{-H})_2\text{Os}_3(\text{CO})_9(\mu_3\text{-}\eta^2\text{-C}{=}\text{CH}_2)$ were detected in the proton NMR spectrum when commercial $\text{BH}_3\cdot\text{THF}$ was employed and the reaction mixture was allowed to remain at room temperature for several days.

Attempted Reduction of $(\mu\text{-H})_2\text{Os}_3(\text{CO})_9(\mu_3\text{-}\eta^2\text{-C}{=}\text{CH}_2)$ by $\text{BH}_3\cdot$THF. An excess of B_2H_6 (0.2758 mmol) was condensed into a THF solution of $(\mu\text{-H})_2\text{Os}_3(\text{CO})_9(\mu_3\text{-}\eta^2\text{-C}{=}\text{CH}_2)$ (0.0208 mmol). The solution was then stirred at room temperature for 12 h. Volatile components were removed and the residue dissolved in CDCl_3. ^{1}H NMR showed no formation of $(\mu\text{-H})_3\text{Os}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$ or other cluster species, only the starting vinylidene cluster, $(\mu\text{-H})_2\text{Os}_3(\text{CO})_9(\mu_3\text{-}\eta^2\text{-C}{=}\text{CH}_2)$. Similar results were observed when the $\text{BH}_3\cdot\text{THF}$ contained 5% NaBH_4 and when commercial $\text{BH}_3\cdot\text{THF}$ was employed and the solutions were allowed to stand at room temperature up to 10 days. On occasion, trace amounts of the ethylidyne cluster, $(\mu\text{-H})_3\text{Os}_3(\text{CO})_9(\mu_3\text{-CCH}_3)$, were detected in the proton NMR spectrum when commercial $\text{BH}_3\cdot\text{THF}$ was employed.

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Supporting Information Available: Tables of crystal data, atomic coordinates, bond distances, and angles, and anisotropic displacement parameters (4 pages). Ordering information is given on any current masthead page.

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